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US Army Research Laboratory

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Weapons and Materials Research Directorate, ARL

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14. ABSTRACT The previous configuration of the laboratory-scale confined laser ignition chamber recently used at the US Army Research Laboratory did not hold pressure up to 70 MPa. This report addresses changes in sealing materials that allow for complete combustion of propellant samples at loading densities up to 0.11 g/cm^3 without the previously encountered failure of the blowout diaphragm or propellant combustion product blow-by at the window seal. Safety considerations for these changes are discussed.			
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1. Introduction

Laboratory-scale confined propellant laser ignition chambers have been used at the US Army Research Laboratory (ARL) for more than 2 decades.[1-5] The laboratory-scale chambers used in these investigations were constructed of steel with a port for a pressure transducer, or of acrylic with no pressure port for time-resolved imaging. All of these chambers had internal volumes less than 1 cm^3, with some of the early chambers having volumes as low as 0.1 cm^3. Designs typical of the chambers used in the 1990s and 2000s are shown in Fig. 1. The pressure transducers used with these chambers were designed for pressures up to 70 MPa, with typical peak pressure measurements around 35 MPa depending on the material being studied, loading density, and thickness of the Mylar* blowout diaphragm used to vent the chamber.

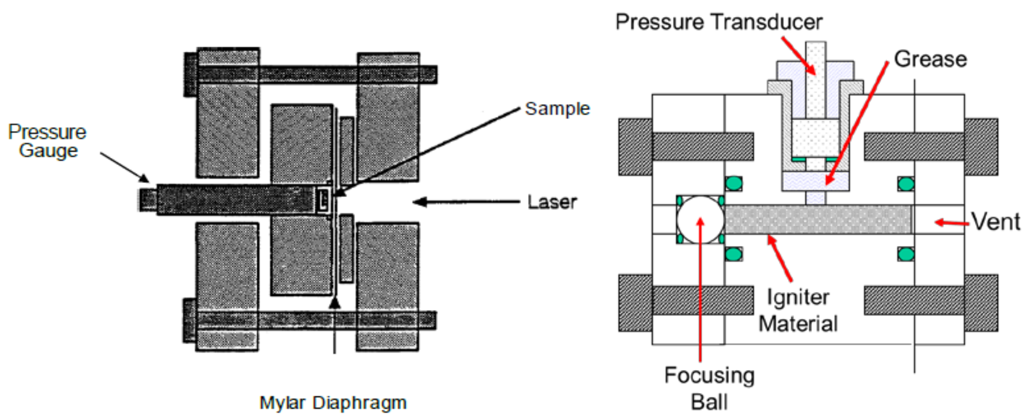

Fig. 1 Typical configurations of laboratory-scale laser ignition chambers used in 1990s–2000s. The chamber on the left is from Forch et al.[1]; the chamber on the right is similar to the one used in Howard et al.[4]

During fiscal year (FY) 2013 Gottfried et al.[5] developed a new laboratory-scale laser ignition chamber that employed a pressure transducer designed for maximum pressure of 100 MPa.[5] The chamber, shown schematically in Fig. 2, shares some design features with a chamber used a decade earlier for 30-mm medium-caliber gun studies. As a comparison, the bore of the chamber on the right side of Fig. 1 was patterned after the flash tube in the family of 30-mm ammunition used with the M230 chain gun. Its internal dimensions were 4.6-mm diameter and 22.9-mm long, resulting in a volume of 380 mm^3. The chamber designed by Gottfried et al. had a larger bore of 6.4-mm diameter and was 15.9-mm long, resulting in a volume of

*Mylar polyester film is a registered trademark of DuPont Teijin Films.

500 mm^3. Both chambers were of 3-piece construction, with a window-retaining flange, the chamber body, and the diaphragm-retaining flange.

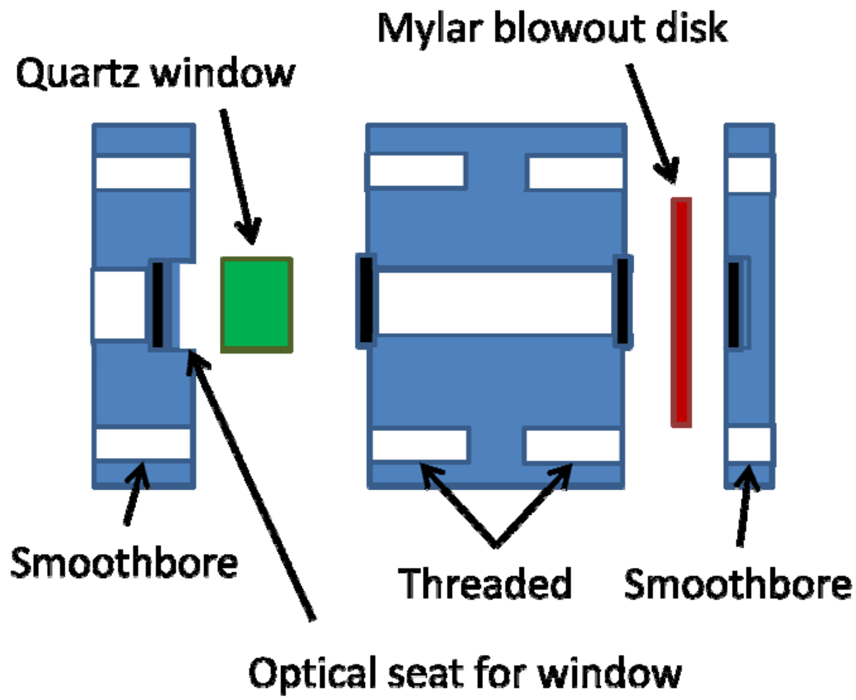

Fig. 2 Schematic of the current laser-ignition chamber. The chamber is from Gottfried et al.[5]

All of the chambers depicted in Figs. 1 and 2 used a Mylar diaphragm as a pressure relief device to vent the chamber. Beyer and Hirlinger noted that a 0.15-mm-thick Mylar diaphragm will rupture at a pressure near 14 MPa.[3] Gottfried et al.[5] observed a linear relationship in the rupture pressure of stacks of 0.08-mm-thick Mylar, with a 0.30-mm-thick stack rupturing at pressures around 17 MPa, and a single piece of 0.33-mm-thick Mylar rupturing at pressures around 24 MPa.[5]

As part of the FY 2017 mission in Disruptive Energetics and Propulsion Technologies, current experiments at ARL employ the chamber designed by Gottfried et al.[5] to investigate high-energy propellants such as JA2 and M10. The goal of the project is to investigate novel laboratory-scale techniques to determine energy content of new high-performance propellant candidates. The current experiments will be used to establish baseline measurements in this chamber using conventional propellants with known performance characteristics. For these propellants, complete combustion peak pressures up to 70 MPa are measured at chamber loading densities of 0.11 g/cm^3 – about 55 mg of propellant. Specifically, we do not want the chamber's blowout diaphragm to function prior to full combustion of the propellant. During the course of these experiments we encountered issues with propellant combustion product blow-by and failure of the diaphragm to hold the target pressure. The remainder of this report addresses modifications to the current laboratory-scale laser ignition chamber to enable it to hold pressure up to 70 MPa. These modifications include changes to the blowout diaphragm, alternate sealing materials, and modifications to the assembly of the cell.

2. Experimental Procedures

A full description of the chamber can be found in Gottfried et al.[5] In addition to the window-retaining flange, the chamber body, and the diaphragm-retaining flange mentioned in the introduction, the original configuration for the chamber included a fused silica parallel window (12.7-mm diameter and 9.52-mm thick), Viton[†] size -012 O-ring seals, and a Mylar blowout diaphragm of various thickness made from combinations of 0.008- and 0.033-cm-thick stock. Figure 3 shows a photo of the unassembled chamber.

Fig. 3 Laboratory-scale laser ignition chamber ready for assembly

A Megawatt Lasers (Hilton Head Island, SC) variable pulse-width, variable energy Nd:YAG laser was used as an ignition source at a wavelength of 1.064 µm. The laser configuration parameters were typically set for a pulse width of 6.295 ms and 300 V for the flashlamp. A small portion of the output from the laser was split off to a laser energy head that was used as a proportional pulse energy monitor. The remainder of the laser pulse was spatially reduced and collimated to a nominal 3-mm diameter using 2 lenses with focal lengths of 1,000 mm and 500 mm configured as a Galilean beam contractor (the combination of temporal pulse

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[†]Viton synthetic rubber and fluoropolymer elastomer is a registered trademark of DuPont Performance Elastomers L.L.C.

length, pulse energy, and the relatively long focal length of the lenses used resulted in a peak intensity at beam waist below threshold for dielectric breakdown of the ambient air). The assembled chamber was mounted to an XYZ stage beneath a turning mirror that directed the laser pulse from horizontal propagation downward vertically through the window of the chamber, with the window retaining flange at the top of the chamber. In general this experimental configuration resulted in 4.8–5.0 J of laser energy transmitted through the laser window into the chamber. A catch can was placed beneath the blowout diaphragm. The chamber was enclosed by a polycarbonate box that was vented to the laboratory exhaust system.

Propellant samples were cut from 6.4-mm-diameter stock and the edges of the propellant slices were trimmed off to give nominally 5- × 5-mm squares. These samples, once loaded into the chamber, were centered with the path of the laser pulse aligned to the center of the bore of the chamber. The propellant samples sat parallel to the blowout diaphragm at the bottom of the chamber and perpendicular to the vertical downward propagation of the laser pulse.

A 100-MPa Kistler (Kistler USA: Amherst, NY) type 601B1 transducer with a type 5010 dual-mode amplifier was used with an Agilent Technologies (Santa Clara, CA) InfiniiVision digital oscilloscope to record the pressure traces. Data was saved from the oscilloscope using a USB thumb drive and was transferred to a CD-ROM via a non-networked laboratory PC. A small amount of silicon grease was applied to the tip of the pressure transducer before each experiment to protect the transducer from damage and minimize thermal effects in the pressure trace.

3. Results and Discussion

3.1 Blowout Diaphragm

While collecting data sets it is essential to ensure no leaks or venting of the chamber. For each individual experiment we want to ensure complete combustion of the propellant and measure a pressure trace that yields a peak pressure consistent with the mass of propellant used. In other words, we do not want the blowout diaphragm to function with a safe loading of propellant. Pressures as great as 70 MPa are well in excess of burst pressures we can get with relatively thick stacks of Mylar diaphragms. The higher pressures (compared to the previous experiments) are desirable to maximize the signal-to-noise ratio of the pressure trace, and to ensure sufficient propellant material for complete combustion following laser ignition. We tried 0.13-, 0.25-, and 0.51-mm-thick brass shim stock as alternates to Mylar as the blowout diaphragm. The thinner 0.13- and 0.25-mm brass shim stocks still ruptured at moderate loadings of propellant. The 0.51-mm brass shim stock held pressure with loading of up to 55-mg JA2 propellant in the chamber. The thicker shim stock did form a bubble or dent at higher peak pressures. In Fig. 4 we measured dent depth on the used shims and found a linear relationship with peak pressure over a range of 30–68 MPa.

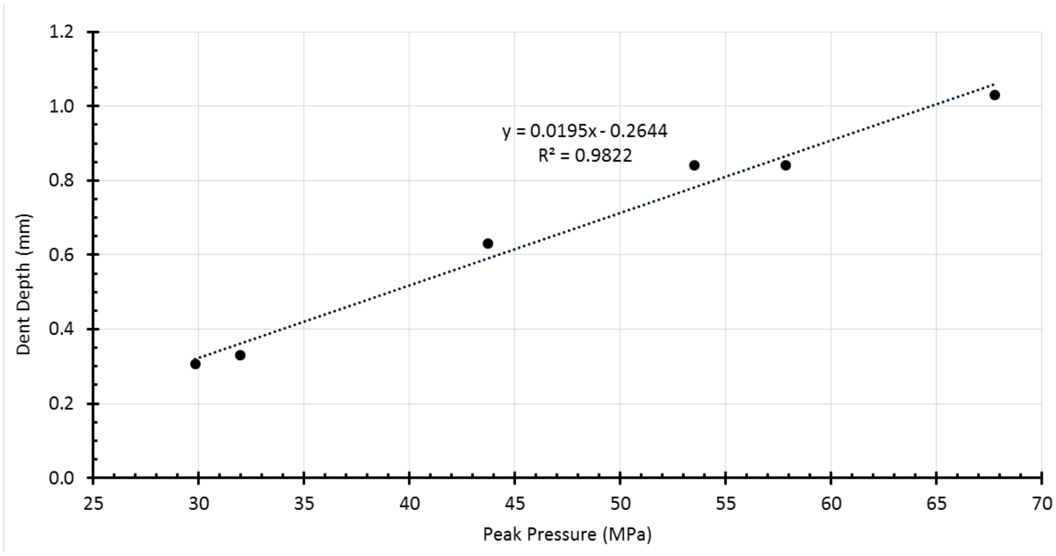

Fig. 4 Dent depth in the 0.51-mm-thick brass shim stock used in place of a Mylar blowout diaphragm as a function of peak pressure. All data shown were generated using JA2 propellant.

In addition to replacing Mylar with 0.51-mm-thick brass, we also reversed the diaphragm-retaining flange so only a single Viton O-ring is used to retain the brass shim on the face of the chamber body. The brass shim stock was cut into 20- × 20-mm squares. The 10-32 machine screws used to secure the flange were backed with steel washers to help distribute the clamping force, and all machine screws were sequentially torqued to 10 lb-in and then 20 lb-in. Finally, the secured flange with the brass shim was visually inspected from all directions to ensure even, parallel spacing from the chamber body. A new O-ring and brass shim was used for each experiment.

3.2 Safety Considerations

The use of the thicker brass shim stock in place of Mylar for the blowout diaphragm results in essentially a non-venting chamber, as we have yet to reach a peak pressure that will fail the 0.51-mm brass shim as a blowout diaphragm. Sample loading density should be limited so that peak pressure does not exceed 70 MPa. From prior experiments, we know a combination shim made from squares of 0.13- and 0.25-mm brass for a total thickness of 0.38 mm will vent at pressures less than 70 MPa. When investigating new propellant samples it is recommended to first use the 0.38-mm-thick brass blowout diaphragm at lower loading densities until the experimental performance characteristics of the new sample are understood prior to proceeding to the use of the thicker 0.51-mm diaphragm.

3.3 Estimation of Chamber Volume Increase from Dent Depth

Using the data in Fig. 4, we can estimate chamber volume expansion if we make the following assumptions: a) the dent can be approximated as a paraboloidal dish, and b) the radius of the dent at its rim is equal to that of the bore of the chamber minus the thickness of the shim. These 2 assumptions leave us with a paraboloidal dish with a rim radius of 2.665 mm and a depth taken from Fig. 4. The volume of a paraboloidal dish can be calculated as 50% of the volume of a right cylinder of the same radius and depth. At the highest pressures indicated in Fig. 4, a dent depth of 1.0 mm corresponds to a chamber volume expansion of about 2.2%. We would expect volume expansion to scale in a linear fashion within the pressure range of the trend line plotted in Fig. 4 down to about 0.7% at the lowest peak pressure of 30 MPa. The estimated change in volume of the cell is small enough that we accept the peak pressure data as generated with this known error.

3.4 Window Sealing

Blow-by of propellant combustion products at the window has been a common problem at elevated pressures. Figure 5 shows an extreme case of blow-by at the window seal. The left side of the photo shows the window side of the chamber body; one can see the brass diaphragm at the bottom of the chamber volume. The right side of the photo shows the window in its flange with a backing O-ring. All of the O-rings used for this experiment were conventional -012 Viton rings with standard round cross section; the cross-sectional diameter of these rings is 1.6 mm. The cell was loaded with 50 mg of JA2 propellant scrap for a loading density of 0.10 g/cm^3. The pressure trace on the oscilloscope indicated a peak pressure of around 40 MPa; the peak pressure is lower than what would be expected for combustion of 50-mg JA2 in this chamber. We believe that the dynamic pressure loading on the window compressed the backing O-ring, which allowed the window to break the seal with the O-ring on the face of the body of the chamber. Once the face seal was broken, the combustion products vented across the gap between the chamber face and the window flange, resulting in a failed experiment. In Fig. 5 the evidence of blow-by is obvious on both metal surfaces. When the photo in Fig. 5 is expanded there is a clear indication that the O-ring on the face seal has been damaged by the flow of propellant combustion products escaping the bore of the chamber.

Fig. 5 **Extreme example of blow-by at the window seal. The chamber was filled with 50 mg of JA2 scrap and leaked due to seal failure at a pressure of about 40 MPa.**

To address the blow-by problem, we investigated combinations of alternate seal materials for the window of the chamber: a) square and X-section rings in place of the standard round-section O-ring, b) soft metal window seat in place of the window backing O-ring, and c) round copper shaft shims around the face O-ring. We did not exhaust all combinations of these materials once we found a solution that worked. The solution that worked best to eliminate blow-by at higher pressures involved the use of 2 solder (50% lead/50% tin) rings as a soft metal window seat in place of the Viton backing O-ring, a Viton X-section O-ring in place of the standard O-ring for the chamber face seal, and a copper shaft shim to limit the compression of the solder rings. Figures 6 and 7, respectively, show schematics of uncompressed and compressed assembly of the window flange to the chamber body.

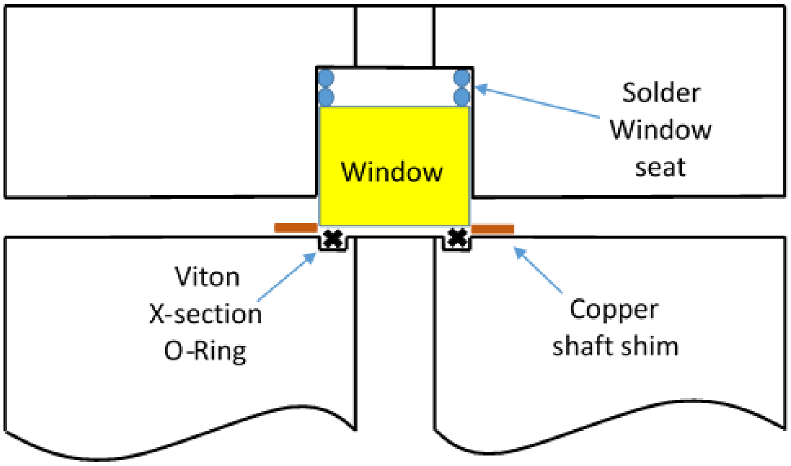

Fig. 6 **Assembly schematic of alternate window sealing materials for the chamber. Relative sizes of the chamber components are not to scale.**

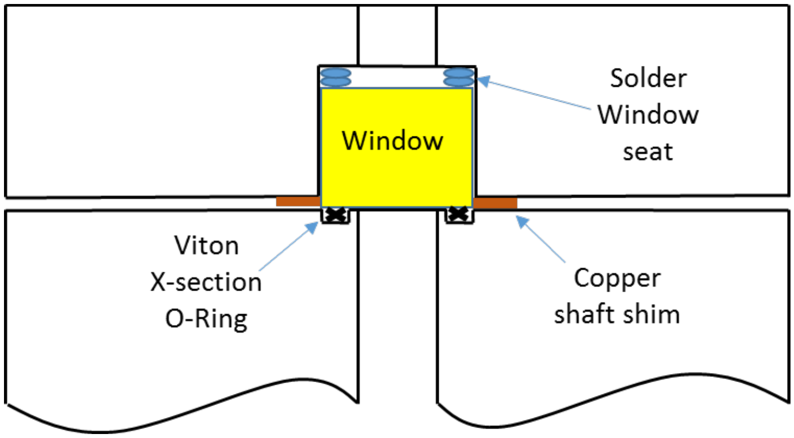

Fig. 7 Schematic of window end of the assembled chamber with machine screws (not indicated) torqued to 20 lb-in. Compression of the solder window seat and X-section O-ring is not to scale.

When the machine screws that hold the chamber together are torqued to 20 lb-in, the window compresses the solder seat and the X-section O-ring. The thickness of the copper shim limits the amount of static compression of the solder seat. Copper shims were purchased in 3 thicknesses: 0.41, 0.25, and 0.13 mm. The thickest copper shim is used first. With each ignition experiment, the dynamic pressure loading on the window further compresses the window seat, so that the seat flattens out more with each experiment and the window sits further in the well in the flange. To maintain a face seal with the X-section O-rings, thinner copper shims are used in subsequent experiments. The X-section O-ring is replaced for each experiment. Once the window has recessed into its well below the surface of the flange the solder seat needs to be replaced, since a) the X-section O-ring may leak even if no copper shim is used, and b) at this point the flattened seat may have extruded into the laser port, partially blocking optical access to the chamber.

Figure 8 shows the fabrication of a solder ring using an O-ring groove in the diaphragm retainer flange. The solder has the same 1.6-mm cross section as the -012 Viton O-ring. The solder is snipped flush with the surface of the flange, which is then turned over and pressed by hand against a flat surface. The solder ring is removed from the flange using a flat blade mini screwdriver. Figure 9 shows a conventional O-ring, an X-section O-ring, and a solder ring side by side for comparison. Two solder rings are used for the seat; the gaps in each ring are offset by 180° when placed in the well in the flange.

Fig. 8 Fabrication of a solder window seat using an O-ring groove in the diaphragm flange

Fig. 9 Conventional O-ring (left), X-section O-ring (center), and solder ring (right). The O-rings, sized -012, have an outer diameter of 12.7 mm and an inner diameter of 9.5 mm.

Figure 10 shows the chamber disassembled following the successful laser ignition of 44.156-mg M10 propellant. No blow-by is evident in the photo. The cloudiness of the window is typical; the window surface is easily cleaned using a couple of drops of methanol. The gap in the upper solder ring is visible through the window at approximately the 4 o'clock position. The flattening of the solder rings is also apparent, as the solder is extruding towards the laser access port in the flange.

Fig. 10 Post-experiment photo of the chamber that had been filled with 44.156-mg M10 propellant. Peak pressure achieved was 48 MPa.

On the right side of Fig. 10, the dent in the brass diaphragm is clearly visible. Blow-by on the diaphragm end of the chamber has never been a significant problem. Any blow-by on this end would be visible on the brass, and none is evident.

Figure 11 is a pressure-time trace from the laser ignition of 42.982 mg of JA2 propellant at a loading density of 0.085 g/cm^3. This was the third experiment with a new pair of solder rings for the window seat. The same window seat was used for 3 additional experiments at lower loading densities of M10 propellant. This series of experiments was conducted to get an indication of how many experiments could be run with the same window seat. At the conclusion of the final experiment in this series, the solder had extruded into a significant cross section of the bore of the chamber. While we were able to run 6 experiments with a pair of solder rings as the window seat, we typically replace the seat after 4 experiments have been run.

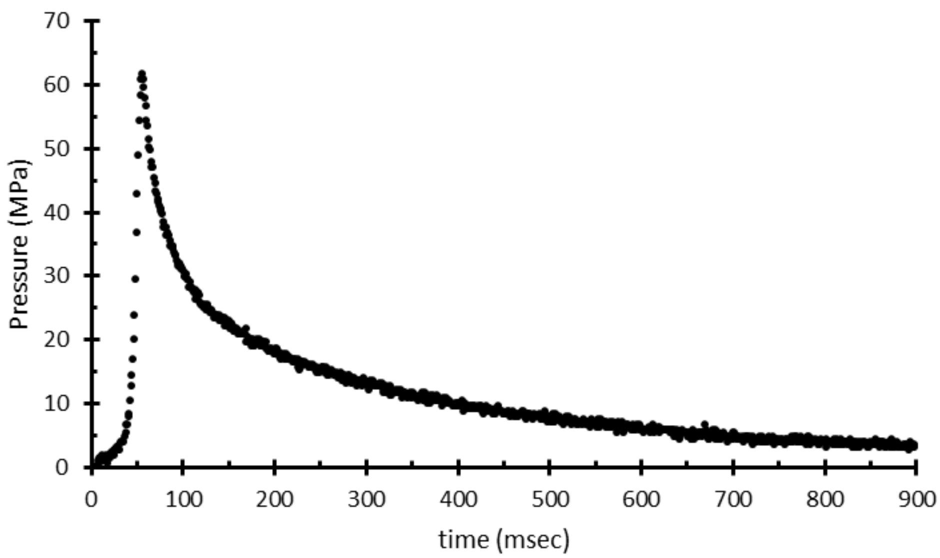

Fig. 11 Pressure-time trace from laser ignition of 42.982-mg JA2 propellant. Peak pressure achieved was 61.8 MPa.

As an alternate to using a solder ring for the window seat, we have also fabricated a seat using a punch set and 1.6-mm-thick lead sheet. Figure 12 shows 2 of these seats, unused and used. A center hole to clear the laser port is first made using a 7-mm punch; this hole is then centered and the seat is punched out from the lead sheet using an 11-mm punch. The shape of the punch results in a raised edge on the outside of the seat. Care is taken when assembling the chamber to center the seat within the well of the window flange. Once this seat is used, it expands to fill the diameter of the window well. We can typically use this lead seat for 2 experiments before it needs to be replaced.

Fig. 12 Lead window seat punched from 1.6-mm-thick lead sheet (left), and used lead window seat (right)

The primary advantage of the lead window seat pictured in Fig. 12 is the continuity of the seat material around the perimeter of the seat. In contrast, a solder ring formed as previously described will have a discontinuity where the solder was cut. The ring discontinuity or gap could lead to uneven dynamic loading to the point of failure of the window the ring supports if only a single ring is used. When using 2 solder rings, with the gaps offset as previously described, we believe the gap in each ring is filled in with material from the other ring when the window assembly is statically compressed during chamber assembly. We have used both types of window seats successfully. The solder rings are preferred over the solid lead seat only due to the more involved and time-consuming process to fabricate a well-centered lead seat using a simple punch.

4. Conclusion

We have successfully addressed sealing issues with the laboratory-scale laser ignition chamber that is currently in use for propellant laser ignition studies at ARL. The useful pressure limit of the chamber was initially increased by replacing the Mylar blowout diaphragm with a 0.51-mm-thick brass shim. At these higher pressures combustion product blow-by became an issue with the original window sealing O-rings. This problem was resolved by replacing the original Viton window backing O-ring with a soft metal seat made from lead/tin solder or punched from lead sheet. The chamber body face seal O-ring for the window was replaced with an X-section O-ring and a copper shaft shim was added to limit static compression of the window seat during assembly. With these changes we have successfully been able to use this chamber at peak pressures approaching 70 MPa.

At higher pressures the brass shim does form a permanent dent, which increases the effective chamber volume for that experiment. We estimate this volume increase at 2.2% for a peak pressure of 68 MPa using JA2 propellant. The estimated change in volume of the cell is small enough that we accept the peak pressure data as generated with this known error.

The use of the thicker brass shim stock in place of Mylar for the blowout diaphragm results in essentially a non-venting chamber, as we have yet to reach a peak pressure that will fail the 0.51-mm brass shim as a blowout diaphragm. Sample loading density should be limited so that peak pressure does not exceed 70 MPa to avoid damaging the pressure transducer. When investigating new propellant samples it is recommended to first use a thinner brass blowout diaphragm at lower loading densities until the experimental performance characteristics of the new sample are understood prior to proceeding to the use of the thicker 0.51-mm diaphragm.

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List of Symbols, Abbreviations, and Acronyms

ARL	US Army Research Laboratory
CD-ROM	compact disc–read-only memory
FY	fiscal year
PC	personal computer
USB	Universal Serial Bus

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